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# (12) United States Patent

Crocker et al.

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### (54) CRYSTAL FORMS OF (-)-6-CHLORO-4-CYCLOPROPYLETHYNYL-4-TRIFLUOROMETHYL-1,4-DIHYDRO-2H-3,1-BENZOXAZIN-2-ONE

(75) Inventors: Louis S. Crocker, Belle Mead, NJ
(US); Joseph L. Kukura, II, Somerset,
NJ (US); Andrew S. Thompson,
Mountainside, NJ (US); Christine
Stelmach, Westfield, NJ (US); Steven
D. Young, Lansdale, PA (US)

(73) Assignee: Merck & Co., Inc., Rahway, NJ (US)

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## Related U.S. Application Data

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| (51) | Int. Cl. <sup>7</sup> C071 | 265/18   |
|------|----------------------------|----------|
| (52) | U.S. Cl                    | . 544/92 |
| (58) | Field of Search            | . 544/92 |

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Primary Examiner—Richard L. Raymond (74) Attorney, Agent, or Firm—Kenneth R. Walton; Valerie J. Camara

## (57) ABSTRACT

The instant invention describes a method for crystallizing (–)-6-chloro-4-cyclopropylethynyl-4-trifluoromethyl-1,4-dihydro-2H-3,1-benzoxazin-2-one from a solvent and antisolvent solvent system and producing the crystalline product. The desired final crystal form, Form I, can be produced when using methanol or ethanol. Form II is isolated from 2-propanol and can be converted to the desired crystal form at low drying temperatures, such as between about a temperature of 40° C. and 50° C.

#### 14 Claims, 7 Drawing Sheets

<sup>\*</sup> cited by examiner